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REMOVAL SUPPORT TEAM 3
EPA CONTRACT EP-S2-14-01

RST 3-04-F-0033

TRANSMITTAL MEMO

To:	Mr. Eric Daly, On-Scene Coordinator Removal Action Branch U.S. EPA, Region II
From:	Smita Sumbaly, Data Reviewer RST 3, Region II
Subject:	Niagara Falls Boulevard Site Data Validation Assessment
Date:	October 26, 2017

The purpose of this memo is to transmit the following information:

- Data validation results for the following parameters:

  TAL Metals and Niobium 5 Samples

- Matrices and Number of Samples

  Soil 5 Samples

- Sampling Date: August 9, 2017

The final data assessment narrative and original analytical data package are attached.

cc:	RST 3 SPM:	Patrick Buster
	RST 3 SITE FILE TDD #:	TO-0010-0005
	RST 3 ANALYTICAL TDD #:	TO-0010-0079
	TASK#:	4079

U.S. ENVIRONMENTAL PROTECTION AGENCY

MEMORANDUM

DATE: October 26, 2017

TO: Mr. Eric Daly, On-Scene Coordinator
U.S. EPA, Region II

FROM: Smita Sumbaly
RST 3 Data Review Team

SUBJECT: **QA/QC Compliance Review Summary**

As requested quality control and performance measures for the data packages noted have been examined and compared to EPA standards for compliance. Measures for the following general areas were evaluated as applicable:

Data Completeness	Holding Time
Calibration, Initial	Calibration, Continuing
Blanks	Laboratory Control Sample
ICP Serial Dilution	Matrix Spike/Matrix Spike Duplicates
ICP Interference Check	ICP-MS Internal Standards
ICP MS Tune Analysis	

Any statistical measures used to support the following conclusions are attached so that the review may be reviewed by others.

Summary of Results

	TAL Metals + Niobium
Acceptable as Submitted	____
Acceptable with Comments	__X__
Unacceptable, Action Pending	____
Unacceptable	____

Data Reviewed by: Smita Sumbaly Date: 10/26/2017

Approved By: _____ Date: 10/26/17

Area Code/Phone No.: (732) 585-4410

NARRATIVE

PCS No. 4079

SITE NAME: **Niagara Falls Boulevard Site**
9524 & 9540 Niagara Falls Boulevard
Niagara Falls, New York

Laboratory Name: TestAmerica Laboratories, Inc., 13715 Rider Trail North, Earth City, MO 63045. Subcontracted by Eberline Analytical Corporation, 601 Scarboro Road, Oak Ridge, Tennessee 37830-7371.

INTRODUCTION:

The laboratory's portion of this case consisted of five soil/rock/slag samples for TAL Metals plus Niobium analyses. The samples were collected on August 9, 2017. The TestAmerica Laboratories, Inc. Job number is: 160-23970-1.

The laboratory reported No problem(s) with the receipt of these samples.

The laboratory reported No QC problems with the analysis of TAL Metals plus Niobium.

The evaluator has commented on the criteria specified under each fraction heading. All criteria have been assessed, but no discussion is given where the evaluator has determined that criteria were adequately performed or require no comment. Details relevant to these comments are given on the following forms.

Appropriate Form Is and Chain of Custody have been copied from the original data package and appended to the data assessment narrative for reference.

Inorganic:

Y Holding Time	Y MS/MSD
Y Calibration, Initial	Y CRQL Check Standard
Y Calibration, Continuing	Y ICP Interference Check
Y Blanks	Y Data Completeness
Y Laboratory Control Sample	Y Serial Dilution
NA Field Duplicate	Y ICP-MS Internal Standards
Y ICP MS Tune analysis	

Comments:

1. Refer to Data Assessment Narrative.

REGION II RST 3 DATA ASSESSMENT REPORT

SITE: Niagara Falls Boulevard Site **SDG No.:** 160-23970-1

LAB: TestAmerica St. Louis, 13715 Rider Trail North, Earth City, MO 63045. Subcontracted by Eberline Analytical Corporation, 601 Scarboro Road, Oak Ridge, Tennessee 37830-7371.

ANALYSIS: Target Analyte List (TAL) Metals plus Niobium
No. of Samples/Matrix: 5-Soil/Rock/Slag

CONTRACTOR: RST 3

The following table summarizes the analytical methods used for the requested analyses and the USEPA, Region II data validation standard operating procedures (SOPs) used for data validation.

Analysis	Analytical Method	Data Validation SOP No.
TAL Metals plus Niobium	SW-846 Methods 6020A	No. HW-3b (Revision 1), September 2016 and Analytical Method

All data were found to be valid and acceptable except those analytes which have been rejected, "R" (unusable). Due to various QC problems some analytes may have been qualified with a "J" (estimated), "N" (presumptive evidence for the presence of the material), "U" (non-detect), or "JN" (presumptive evidence for the presence of the material at an estimated value) flag. All action is detailed on the attached sheets.

The "R" flag means that the associated value is unusable. In other words, significant data bias is evident and the reported analyte concentration is unreliable.

Reviewer
Signature: Smita Sumbaly **Date:** 10/26/2017

Verified By: _____ **Date:** 10/26/2017

On August 9, 2017, USEPA, Region II, RST 3 personnel collected five soil/rock/slag samples, for TAL metals plus niobium analyses from Niagara Falls Boulevard Site located at 9524 & 9540 Niagara Falls Boulevard, Niagara Falls, New York. These samples were delivered by Federal Express under chain of custody for the requested analyses to Eberline Analytical Corporation, 601 Scarboro Road, Oak Ridge, Tennessee 37830-7371. Eberline Analytical Corporatin ran the samples through their jaw crusher to reduce the sample size and, on August 14, 2017, shipped the samples for TAL Metals plus niobium analyses to TestAmerica St. Louis laboratory located at 13715 Rider Trail North, Earth City, MO 63045. The laboratory verified that samples were received intact, and properly sealed. Sample cooler temperature measured at 17.0 °C.

Field Sample ID	Lab Sample ID	Matrix	Analysis	Sampling Date
SDG 160-23970-1				
NFB-SLAG-01-170809	160-23970-1	Soil/Rock/Slag	TAL Metals plus Niobium	8/09/2017
NFB-SLAG-02-170809	160-23970-2	Soil/Rock/Slag	TAL Metals plus Niobium	8/09/2017
NFB-SLAG-03-170809	160-23970-3	Soil/Rock/Slag	TAL Metals plus Niobium	8/09/2017
NFB-SLAG-04-170809	160-23970-4	Soil/Rock/Slag	TAL Metals plus Niobium	8/09/2017
NFB-SLAG-05-170809	160-23970-5	Soil/Rock/Slag	TAL Metals plus Niobium	8/09/2017

ANALYSIS: TAL METALS + NIOBIUM -ICP MS

The current SOPs HW-3B (Revision 1) July 2015, USEPA Region II for the evaluation of metals data generated through SW 846 Method 6020A, and the National Functional Guidelines for Superfund Inorganic Methods Data Review, January 2017 (based on SOW02.4) were followed for data qualifications.

1. HOLDING TIME AND PRESERVATION

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time or pH (aqueous samples) is not within the acceptable range, the data may not be valid. Those analytes detected in the samples whose holding time (180 days) or pH (<2) have not been met will be qualified as estimated, "J"; the non-detects will be flagged as unusable, "R". Qualifications were applied to the samples and analytes as shown below.

Laboratory received the samples at 17^0C, no action were taken.

2. CALIBRATION

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for the metals on the Inorganic Target Analyte List (TAL). Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.

A) INITIAL CALIBRATION

A blank and at least five calibration standards shall be used to establish each analytical

curve. At least one of these standards shall be at or below the CRQL. The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The curve must have a correlation coefficient ≥ 0.995. The percent differences calculated for all of the non-zero standards must be within ±30% of the true value of the standard. The y-intercept of the curve must be less than the CRQL. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

B) INITIAL AND CONTINUING CALIBRATION VERIFICATION
Immediately after each system has been calibrated, the accuracy of the initial calibration must be verified and documented for each target analyte by the analysis of an ICV solution(s).

The CCV standard shall be analyzed at a frequency of every two hours during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample. The percent recovery acceptable limits for ICV/CCV are 90 – 110%. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

3. BLANK CONTAMINATION
Quality assurance (QA) blanks, i.e., method, field, or rinse blanks are prepared to identify any contamination, which may have been introduced into the samples during sample preparation or field activity. Initial calibration blank and continuing calibration blank (ICB and CCB) are used to ensure a stable instrument baseline before and during the analysis of analytical samples. The preparation blank is used to assess the level of contamination introduced to the analytical samples throughout the sample preparation process. Field and rinse blanks measure cross-contamination of samples during field operations. Qualifications were applied to the samples and analytes as shown below.

The following samples have analyte results less than or equal to CRQLs. The associated CCB analyte results are less than or equal to CRQLs. Detects are qualified as U. Sample results are reported at CRQLs.

No problems were found for this criterion.

4. INTERFERENCE CHECK SAMPLE
The Interference Check Sample (ICS) verifies the analytical instrument's ability to overcome interferences typical of those found in samples. The laboratory should have analyzed and reported ICS results for all elements being reported from the analytical run and for all interferents (target and non-target) for these reported elements. The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. Results for the analysis of ICS Solution must fall within the control limits of ± 20% or +2X CRQL (whichever is greater) of the true value for the analytes and interferents included in the

solution. If results that are ≥ MDL are observed for analytes that are not present in the ICS solution, the possibility of false positives exists. If negative results are observed for analytes that are not present in the ICS solution, and their absolute value is ≥ MDL, the possibility of false negatives in the samples exists. In general, ICP sample data can be accepted if the concentrations of Aluminum, Calcium, Iron, and Magnesium in the sample are found to be less than or equal to their respective concentrations in the ICS. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

5. SPIKE SAMPLE ANALYSIS

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The spike Percent Recovery (%R) shall be within the established acceptance limits of 75 – 125%. However, spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. For a matrix spike analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the matrix spike sample.

The laboratory performed the MS/MSD analyses on sample NFB-SLAG-01-170809. The percent recoveries were less than the lower control limit for antimony, cobalt, and sodium. The results reported for sample NFB-SLAG-01-170809 were estimated with a low bias (J- or UJ) for antimony, cobalt, and sodium.

The laboratory performed the MS/MSD analyses on sample NFB-SLAG-01-170809. The percent recoveries were greater than the upper control limit for iron, niobium, and selenium. The relative percent difference (RPD) was also greater than QC limit for niobium. The results reported for sample NFB-SLAG-01-170809 were estimated with a high bias (J+) for iron, niobium, and selenium.

The matrix spike was within control limits for chromium and copper, and RPDs were above the QC limits for chromium, copper and magnesium. These results reported for sample NFB-SLAG-01-170809 were estimated with no bias (J) because the matrix spike recoveries were in compliance.

Sample NFB-SLAG-01-170809 was used for the MS/MSD analyses which yielded non-compliant recoveries for aluminum, barium, beryllium, calcium, magnesium, and manganese. Since the aluminum, barium, beryllium, calcium, magnesium, and manganese concentrations in the source sample were more than four times the added concentrations, data qualifications were not required.

6. DUPLICATE SAMPLE ANALYSIS

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. A control limit of 20% (for aqueous sample) for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit

of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. For a duplicate sample analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the duplicate sample.

Note: Instead of laboratory duplicate analysis, laboratory analyzed matrix spike, matrix spike duplicate and calculated relative percent difference to assess precision.

7. FIELD DUPLICATE

Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the 2 x CRQL for solid matrix shall be used if either the sample or duplicate value is < 5x the CRQL. For field duplicates analysis that does not meet the technical criteria, the action was applied to only the field sample and it's duplicate.

Not Applicable

8. LABORATORY CONTROL SAMPLE

The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. Aqueous/water, soil/sediment, wipe, and filter LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples. All LCS Percent Recoveries (%R) must fall within the control limits of 70-130%, except for Sb and Ag which must fall within the control limits of 50-150%. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

9. ICP SERIAL DILUTION

The serial dilution of samples quantitated by Inductively Coupled Plasma determines whether or not significant physical or chemical interferences exist due to sample matrix. If the analyte concentration is sufficiently high [concentration in the original sample is > 50 times (50x) the Method Detection Limit (MDL)], the Percent Difference (%D) between the original determination and the serial dilution analysis (a five-fold dilution) after correction for dilution shall be less than 15. For a serial dilution analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the serial dilution sample.

No problems were found for this criterion.

10. ICP-MS TUNE ANALYSIS

The Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) tune serves as an initial demonstration of instrument stability and precision. Prior to calibration, the laboratory shall analyze or scan the ICP-MS tuning solution at least five times (5x) consecutively. The tuning solution contains 100 µg/L of Be, Mg, Co, In, and Pb. The solution shall contain all

required isotopes of the above elements. The laboratory shall make any adjustments necessary to bring peak width within the instrument manufacturer's specifications and adjust mass resolution to within 0.1 u over the range of 6-210 u. The Percent Relative Standard Deviation (%RSD) of the absolute signals for all analytes in the tuning solution must be < 5%. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

11. ICP-MS INTERNAL STANDARDS

The analysis of Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) internal standards determines the existence and magnitude of instrument drift and physical interferences. The criteria for evaluation of internal standard results apply to all analytical and Quality Control (QC) samples analyzed during the run, beginning with the calibration. All samples analyzed during a run, with the exception of the ICP-MS tune, shall contain internal standards. A minimum of five internal standards shall be added to each sample. The laboratory shall monitor the same internal standards throughout the entire analytical run and shall assign each analyte to at least one internal standard. The Percent Relative Intensity (%RI) in the sample shall fall within 60-125% of the response in the calibration blank. If the %RI of the response in the sample falls outside of these limits, the laboratory shall reanalyze the original sample at a two-fold dilution with internal standard added. Qualifications were applied to the samples and analytes as shown below.

The following samples have internal standard area response less than expanded minimum criteria. Detects are qualified as estimated J.

For analysis batch 324791, the percent relative intensity (%RI) was greater than the upper control limit for internal standards Sc/2 and Sc/3 in sample mentioned below. Using professional judgment, the associated result reported for calcium in samples NFB-SLAG-03-170809 was estimated (J).

Calcium – J – NFB-SLAG-03-170809

12. PERCENT SOLIDS

The laboratory is required to perform the percent solids determination prior to sample preparation and analysis. All results of a sample with percent solids less than 50% are qualified estimated, "J". Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

13. OTHER PROBLEMS: The temperature of sample shipping cooler containing all samples was 17°C upon receipt at the lab, greater than 4^0C $\pm$ 2^0C. Using professional judgment, qualification was not applied.

14. DILUTIONS, RE-EXTRACTIONS & REANALYSIS:

Samples may be re-analyzed for dilution, re-extraction and for other QC reasons. In such cases, the best result values are consolidated in one single report and the other report is marked as not to be used. The following sample results were reported from a dilution analysis.

20-Fold dilutions: All samples were diluted to bring the concentration of target analytes within the calibration range, except for samples below that were further diluted due to the nature of sample matrix.

50-Fold dilutions:
Aluminum, Calcium, Magnesium, Potassium, Sodium, and Vanadium – NFB-SLAG-01-170809 and NFB-SLAG-03-170809

200-Fold dilutions:
Manganese – NFB-SLAG-04-170809

OTHER ANALYTES WORK TABLE

PROJECT: Niagara Falls Boulevard Site

SAMPLING DATE: August 9 , 2017

SAMPLE #/CONCENTRATION (mg/Kg)

Matrix:	Soil	Soil	Soil	Soil	Soil
Field Sample ID	NFB-SLAG-01-170809	NFB-SLAG-02-170809	NFB-SLAG-03-170809	NFB-SLAG-04-170809	NFB-SLAG-05-170809
Lab Sample ID	160-23970-1	160-23970-2	160-23970-3	160-23970-4	160-23970-5
Sample Weight (g)	0.5389	0.5611	0.5160	0.5198	0.5439
% Solid	98.2	99.8	99.3	100	99.9
Dilution Factor	20, *50	20	20, *50	20, *200	20
Inorganics					
Aluminum	*32000	56000	*62000	53000	89000
Antimony	1.9 UJ-	1.8 U	4.5 J	1.9 U	1.8 U
Arsenic	9.6	3.6 U	16	6.3 J	3.7 U
Barium	1500	180	740	9500	980
Beryllium	53	3.8	51	19	10
Cadmium	2.0	0.39 J	2.8	0.23 U	0.61
Calcium	*190000	120000	*280000 J	150000	140000
Chromium	35 J	700	190	24	160
Cobalt	7.9 J-	20	2.9	0.72 U	0.80 J
Copper	19 J	60	2100	6.1 J	93
Iron	650 J+	1500	6500	190	640
Lead	2.3 J	6.4	220	1.2 U	9.3
Magnesium	*14000 J	42000	*15000	9600	20000
Manganese	1400	1200	3500	*57000	560
Nickel	4.2 J	19	16	1.9 U	1.8 U
Niobium	11 J+	38	66	12 U	46
Potassium	*94 U	190	*320	6700	37 U
Selenium	27 J+	7.3	54	14	14
Silver	5.2	1.0 J	7.5	0.72 U	1.7 J
Sodium	*330 J-	8800	*460 J	940	950
Thallium	1.9 U	1.8 U	2.0 U	1.9 U	1.8 U
Vanadium	*9.4 U	7.6 J	*9.9 J	3.8 U	3.7 U
Zinc	19 U	18 U	110	19 U	18 U

U - non-detected analyte

UJ - not detected at the estimated method detection limit

J - estimated value

J+ - estimated value for potential high bias

J- - estimated value for potential low bias

CASE NARRATIVE

Client: Eberline Analytical Corporation

Project: TAL Metals + Nb

Report Number: 160-23970-1

With the exceptions noted as flags or footnotes, standard analytical protocols were followed in the analysis of the samples and no problems were encountered or anomalies observed. In addition all laboratory quality control samples were within established control limits, with any exceptions noted below. Each sample was analyzed to achieve the lowest possible reporting limit within the constraints of the method. In some cases, due to interference or analytes present at high concentrations, samples were diluted. For diluted samples, the reporting limits are adjusted relative to the dilution required.

TestAmerica St. Louis attests to the validity of the laboratory data generated by TestAmerica facilities reported herein. All analyses performed by TestAmerica facilities were done using established laboratory SOPs that incorporate QA/QC procedures described in the application methods. TestAmerica's operations groups have reviewed the data for compliance with the laboratory QA/QC plan, and data have been found to be compliant with laboratory protocols unless otherwise noted below.

The test results in this report meet all NELAP requirements for parameters for which accreditation is required or available. Any exceptions to NELAP requirements are noted in this report. Pursuant to NELAP, this report may not be reproduced, except in full, without the written approval of the laboratory.

Calculations are performed before rounding to avoid round-off errors in calculated results.

All holding times were met and proper preservation noted for the methods performed on these samples, unless otherwise detailed in the individual sections below.

All solid sample results for Chemistry analyses are reported on an "as received" basis unless otherwise indicated by the presence of a % solids value in the method header. All soil/sediment sample results for radiochemistry analyses are based upon sample as dried and disaggregated with the exception of tritium, carbon-14, and iodine-129 by gamma spectroscopy unless requested as wet weight by the client."

This laboratory report is confidential and is intended for the sole use of TestAmerica and its client.

RECEIPT
The samples were received on 08/15/2017; the samples arrived in good condition, properly preserved. The temperature of the coolers at receipt was 17.0 C.

METALS (ICPMS)
Samples NFB-SLAG-01-170809 (160-23970-1), NFB-SLAG-02-170809 (160-23970-2), NFB-SLAG-03-170809 (160-23970-3), NFB-SLAG-04-170809 (160-23970-4) and NFB-SLAG-05-170809 (160-23970-5) were analyzed for metals (ICPMS) in accordance with EPA SW-846 Methods 6020A. The samples were prepared on 08/21/2017 and analyzed on 08/25/2017, 08/26/2017 and 08/28/2017.

Batch 324638
The following samples were diluted to bring the concentration of target analytes within the calibration range: NFB-SLAG-01-170809 (160-23970-1), NFB-SLAG-02-170809 (160-23970-2), NFB-SLAG-03-170809 (160-23970-3), NFB-SLAG-04-170809 (160-23970-4), NFB-SLAG-05-170809 (160-23970-5), (160-23970-A-1-C MS), (160-23970-A-1-D MSD) and (160-23970-A-1-B SD). Elevated reporting limits (RLs) are provided.

The MS (MSD) recovery and precision for niobium is outside the established QC limits. The MS/MSD is a multiple element spiking solution. The majority of elements in the spiking solutions are either within acceptable criteria or the concentration is four times greater than the spiked amount, making recovery unreliable. This indicates that a matrix interference is present in the sample. Method performance is demonstrated by acceptable LCS recovery. No further action is required. (160-23970-A-1-C MS) and (160-23970-A-1-D MSD)

Batch 324640
Method(s) 6020A: Due to the high concentration of Barium, Manganese, and Beryllium the matrix spike / matrix spike duplicate (MS/MSD) for preparation batch 160-323386 and analytical batch 160-324640 could not be evaluated for accuracy and precision. The associated laboratory control sample (LCS) met acceptance criteria.(160-23970-A-1-C MS) and (160-23970-A-1-D MSD)

The matrix spike / matrix spike duplicate (MS/MSD) recoveries for preparation batch 160-323386 and analytical batch 160-324640 were outside control limits for Cobalt, Iron, Antimony, and Selenium. Sample matrix interference and/or non-homogeneity are suspected because the associated laboratory control sample (LCS) recovery was within acceptance limits.(160-23970-A-1-C MS) and

(160-23970-A-1-D MSD)

The following samples were diluted due to the nature of the sample matrix. Samples cause internal standard failures when ran at a lesser dilution: NFB-SLAG-01-170809 (160-23970-1), NFB-SLAG-02-170809 (160-23970-2), NFB-SLAG-03-170809 (160-23970-3), NFB-SLAG-04-170809 (160-23970-4), NFB-SLAG-05-170809 (160-23970-5), (160-23970-A-1-C MS), (160-23970-A-1-D MSD) and (160-23970-A-1-B SD). Elevated reporting limits (RLs) are provided.

The MS (MSD) recovery and precision for Chromium and Copper is outside the established QC limits. The MS/MSD is a multiple element spiking solution. The majority of the other elements in the spiking solution are either within acceptable criteria or the concentration is four times greater than the spiked amount, making recovery unreliable. This indicates that a matrix interference is present in the sample. Method performance is demonstrated by acceptable LCS recovery. No further action is required. (160-23970-A-1-C MS) and (160-23970-A-1-D MSD)

Batch 324791
The following sample was diluted to bring the concentration of target analytes within the calibration range: NFB-SLAG-04-170809 (160-23970-4). Elevated reporting limits (RLs) are provided.

The following samples were diluted due to the nature of the sample matrix. Samples caused internal standard failures when ran at a lesser dilution: NFB-SLAG-01-170809 (160-23970-1), NFB-SLAG-03-170809 (160-23970-3), (160-23970-A-1-C MS), (160-23970-A-1-D MSD) and (160-23970-A-1-B SD). Elevated reporting limits (RLs) are provided.

Due to the high concentration of Aluminium, Calcium, and Magnesium the matrix spike / matrix spike duplicate (MS/MSD) for preparation batch 160-323386 and analytical batch 160-324791 could not be evaluated for accuracy and precision. The associated laboratory control sample (LCS) met acceptance criteria.(160-23970-A-1-C MS) and (160-23970-A-1-D MSD)

The matrix spike / matrix spike duplicate (MS/MSD) recoveries for preparation batch 160-323386 and analytical batch 160-324791 were outside control limits for Sodium. Sample matrix interference and/or non-homogeneity are suspected because the associated laboratory control sample (LCS) recovery was within acceptance limits.(160-23970-A-1-C MS) and (160-23970-A-1-D MSD)

No additional analytical or quality issues were noted, other than those described above or in the Definitions/Glossary page.

PERCENT SOLIDS
Samples NFB-SLAG-01-170809 (160-23970-1), NFB-SLAG-02-170809 (160-23970-2), NFB-SLAG-03-170809 (160-23970-3), NFB-SLAG-04-170809 (160-23970-4) and NFB-SLAG-05-170809 (160-23970-5) were analyzed for percent solids in accordance with EPA Method 160.3 MOD. The samples were analyzed on 08/16/2017.

No analytical or quality issues were noted, other than those described above or in the Definitions/Glossary page.

Method Summary

Client: Eberline Analytical Corporation
Project/Site: TAL Metals + Nb

TestAmerica Job ID: 160-23970-1

Method	Method Description	Protocol	Laboratory
6020A	Metals (ICP/MS)	SW846	TAL SL
Moisture	Percent Moisture	EPA	TAL SL

Protocol References:

EPA = US Environmental Protection Agency

SW846 = "Test Methods For Evaluating Solid Waste, Physical/Chemical Methods", Third Edition, November 1986 And Its Updates.

Laboratory References:

TAL SL = TestAmerica St. Louis, 13715 Rider Trail North, Earth City, MO 63045, TEL (314)298-8566

Accreditation/Certification Summary

Client: Eberline Analytical Corporation
Project/Site: TAL Metals + Nb

TestAmerica Job ID: 160-23970-1

Laboratory: TestAmerica St. Louis

Unless otherwise noted, all analytes for this laboratory were covered under each accreditation/certification below.

Authority	Program	EPA Region	Identification Number	Expiration Date
Louisiana	NELAP	6	04080	06-30-18

The following analytes are included in this report, but are not accredited/certified under this accreditation/certification:

Analysis Method	Prep Method	Matrix	Analyte
6020A	3050B	Solid	Niobium

The following analytes are included in this report, but accreditation/certification is not offered by the governing authority:

Analysis Method	Prep Method	Matrix	Analyte
Moisture		Solid	Percent Moisture
Moisture		Solid	Percent Solids

Qualifiers

Metals

Qualifier	Qualifier Description
F1	MS and/or MSD Recovery is outside acceptance limits.
F2	MS/MSD RPD exceeds control limits
J	Result is less than the RL but greater than or equal to the MDL and the concentration is an approximate value.
4	MS, MSD: The analyte present in the original sample is greater than 4 times the matrix spike concentration; therefore, control limits are not applicable.
^	Instrument related QC is outside acceptance limits.

Glossary

Abbreviation	These commonly used abbreviations may or may not be present in this report.
¤	Listed under the "D" column to designate that the result is reported on a dry weight basis
%R	Percent Recovery
CFL	Contains Free Liquid
CNF	Contains No Free Liquid
DER	Duplicate Error Ratio (normalized absolute difference)
Dil Fac	Dilution Factor
DL	Detection Limit (DoD/DOE)
DL, RA, RE, IN	Indicates a Dilution, Re-analysis, Re-extraction, or additional Initial metals/anion analysis of the sample
DLC	Decision Level Concentration (Radiochemistry)
EDL	Estimated Detection Limit (Dioxin)
LOD	Limit of Detection (DoD/DOE)
LOQ	Limit of Quantitation (DoD/DOE)
MDA	Minimum Detectable Activity (Radiochemistry)
MDC	Minimum Detectable Concentration (Radiochemistry)
MDL	Method Detection Limit
ML	Minimum Level (Dioxin)
NC	Not Calculated
ND	Not Detected at the reporting limit (or MDL or EDL if shown)
PQL	Practical Quantitation Limit
QC	Quality Control
RER	Relative Error Ratio (Radiochemistry)
RL	Reporting Limit or Requested Limit (Radiochemistry)
RPD	Relative Percent Difference, a measure of the relative difference between two points
TEF	Toxicity Equivalent Factor (Dioxin)
TEQ	Toxicity Equivalent Quotient (Dioxin)

1A-IN
INORGANIC ANALYSIS DATA SHEET
METALS

Client Sample ID: NFB-SLAG-01-170809 | Lab Sample ID: 160-23970-1

Lab Name: TestAmerica St. Louis | Job No.: 160-23970-1

SDG ID.:

Matrix: Solid | Date Sampled: 08/09/2017 12:00

Reporting Basis: DRY | Date Received: 08/15/2017 08:30

% Solids: 98.2

CAS No.	Analyte	Result	RL	MDL	Units	C	Q	DIL	Method
7429-90-5	Aluminum	32000	120	47	mg/Kg			50	6020A
7440-36-0	Antimony	ND	4.7	1.9	mg/Kg		~~F1~~ J-	20	6020A
7440-38-2	Arsenic	9.6	9.4	3.8	mg/Kg			20	6020A
7440-39-3	Barium	1500	19	4.7	mg/Kg			20	6020A
7440-41-7	Beryllium	53	0.94	0.38	mg/Kg			20	6020A
7440-43-9	Cadmium	2.0	0.47	0.23	mg/Kg			20	6020A
7440-70-2	Calcium	190000	1200	240	mg/Kg			50	6020A
7440-47-3	Chromium	35	9.4	4.3	mg/Kg		~~F2 F1~~ J	20	6020A
7440-48-4	Cobalt	7.9	1.9	0.71	mg/Kg		~~F1~~ J-	20	6020A
7440-50-8	Copper	19	9.4	3.8	mg/Kg		~~F2 F1~~ J	20	6020A
7439-89-6	Iron	650	47	19	mg/Kg		~~F1~~ J+	20	6020A
7439-92-1	Lead	2.3	2.8	1.2	mg/Kg	J		20	6020A
7439-95-4	Magnesium	14000	1200	590	mg/Kg		~~F2~~ J	50	6020A
7439-96-5	Manganese	1400	4.7	1.9	mg/Kg			20	6020A
7440-02-0	Nickel	4.2	4.7	1.9	mg/Kg	J		20	6020A
7440-03-1	Niobium	11	24	11	mg/Kg	J	~~F2 F1~~ J+	20	6020A
7440-09-7	Potassium	ND	240	94	mg/Kg			50	6020A
7782-49-2	Selenium	27	4.7	3.0	mg/Kg		~~F1~~ J+	20	6020A
7440-22-4	Silver	5.2	1.9	0.71	mg/Kg			20	6020A
7440-23-5	Sodium	330	470	180	mg/Kg	J	~~F1~~ J-	50	6020A
7440-28-0	Thallium	ND	4.7	1.9	mg/Kg			20	6020A
7440-62-2	Vanadium	ND	24	9.4	mg/Kg			50	6020A
7440-66-6	Zinc	ND	47	19	mg/Kg			20	6020A

10/11/17

1A-IN
INORGANIC ANALYSIS DATA SHEET
METALS

Client Sample ID: NFB-SLAG-02-170809 Lab Sample ID: 160-23970-2

Lab Name: TestAmerica St. Louis Job No.: 160-23970-1

SDG ID.:

Matrix: Solid Date Sampled: 08/09/2017 12:00

Reporting Basis: DRY Date Received: 08/15/2017 08:30

% Solids: 99.8

CAS No.	Analyte	Result	RL	MDL	Units	C	Q	DIL	Method
7429-90-5	Aluminum	56000	45	18	mg/Kg			20	6020A
7440-36-0	Antimony	ND	4.5	1.8	mg/Kg			20	6020A
7440-38-2	Arsenic	ND	8.9	3.6	mg/Kg			20	6020A
7440-39-3	Barium	180	18	4.5	mg/Kg			20	6020A
7440-41-7	Beryllium	3.8	0.89	0.36	mg/Kg			20	6020A
7440-43-9	Cadmium	0.39	0.45	0.21	mg/Kg	J		20	6020A
7440-70-2	Calcium	120000	450	89	mg/Kg			20	6020A
7440-47-3	Chromium	700	8.9	4.0	mg/Kg			20	6020A
7440-48-4	Cobalt	20	1.8	0.67	mg/Kg			20	6020A
7440-50-8	Copper	60	8.9	3.6	mg/Kg			20	6020A
7439-89-6	Iron	1500	45	18	mg/Kg			20	6020A
7439-92-1	Lead	6.4	2.7	1.1	mg/Kg			20	6020A
7439-95-4	Magnesium	42000	450	220	mg/Kg			20	6020A
7439-96-5	Manganese	1200	4.5	1.8	mg/Kg			20	6020A
7440-02-0	Nickel	19	4.5	1.8	mg/Kg			20	6020A
7440-03-1	Niobium	38	22	11	mg/Kg			20	6020A
7440-09-7	Potassium	190	89	36	mg/Kg			20	6020A
7782-49-2	Selenium	7.3	4.5	2.9	mg/Kg			20	6020A
7440-22-4	Silver	1.0	1.8	0.67	mg/Kg	J		20	6020A
7440-23-5	Sodium	8800	180	67	mg/Kg			20	6020A
7440-28-0	Thallium	ND	4.5	1.8	mg/Kg			20	6020A
7440-62-2	Vanadium	7.6	8.9	3.6	mg/Kg	J		20	6020A
7440-66-6	Zinc	ND	45	18	mg/Kg			20	6020A

1A-IN
INORGANIC ANALYSIS DATA SHEET
METALS

Client Sample ID: NFB-SLAG-03-170809 Lab Sample ID: 160-23970-3

Lab Name: TestAmerica St. Louis Job No.: 160-23970-1

SDG ID.:

Matrix: Solid Date Sampled: 08/09/2017 12:00

Reporting Basis: DRY Date Received: 08/15/2017 08:30

% Solids: 99.3

CAS No.	Analyte	Result	RL	MDL	Units	C	Q	DIL	Method
7429-90-5	Aluminum	62000	120	49	mg/Kg			50	6020A
7440-36-0	Antimony	4.5	4.9	2.0	mg/Kg	J		20	6020A
7440-38-2	Arsenic	16	9.8	3.9	mg/Kg			20	6020A
7440-39-3	Barium	740	20	4.9	mg/Kg			20	6020A
7440-41-7	Beryllium	51	0.98	0.39	mg/Kg			20	6020A
7440-43-9	Cadmium	2.8	0.49	0.23	mg/Kg			20	6020A
7440-70-2	Calcium	280000	1200	240	mg/Kg		J	50	6020A
7440-47-3	Chromium	190	9.8	4.4	mg/Kg			20	6020A
7440-48-4	Cobalt	2.9	2.0	0.73	mg/Kg			20	6020A
7440-50-8	Copper	2100	9.8	3.9	mg/Kg			20	6020A
7439-89-6	Iron	6500	49	20	mg/Kg			20	6020A
7439-92-1	Lead	220	2.9	1.2	mg/Kg			20	6020A
7439-95-4	Magnesium	15000	1200	610	mg/Kg			50	6020A
7439-96-5	Manganese	3500	4.9	2.0	mg/Kg			20	6020A
7440-02-0	Nickel	16	4.9	2.0	mg/Kg			20	6020A
7440-03-1	Niobium	66	24	12	mg/Kg			20	6020A
7440-09-7	Potassium	320	240	98	mg/Kg			50	6020A
7782-49-2	Selenium	54	4.9	3.1	mg/Kg			20	6020A
7440-22-4	Silver	7.5	2.0	0.73	mg/Kg			20	6020A
7440-23-5	Sodium	460	490	180	mg/Kg	J		50	6020A
7440-28-0	Thallium	ND	4.9	2.0	mg/Kg			20	6020A
7440-62-2	Vanadium	9.9	24	9.8	mg/Kg	J		50	6020A
7440-66-6	Zinc	110	49	20	mg/Kg			20	6020A

10/11/17

1A-IN
INORGANIC ANALYSIS DATA SHEET
METALS

Client Sample ID: NFB-SLAG-04-170809 Lab Sample ID: 160-23970-4

Lab Name: TestAmerica St. Louis Job No.: 160-23970-1

SDG ID.:

Matrix: Solid Date Sampled: 08/09/2017 12:00

Reporting Basis: DRY Date Received: 08/15/2017 08:30

% Solids: 100.0

CAS No.	Analyte	Result	RL	MDL	Units	C	Q	DIL	Method
7429-90-5	Aluminum	53000	48	19	mg/Kg			20	6020A
7440-36-0	Antimony	ND	4.8	1.9	mg/Kg			20	6020A
7440-38-2	Arsenic	6.3	9.6	3.8	mg/Kg	J		20	6020A
7440-39-3	Barium	9500	19	4.8	mg/Kg			20	6020A
7440-41-7	Beryllium	19	0.96	0.38	mg/Kg			20	6020A
7440-43-9	Cadmium	ND	0.48	0.23	mg/Kg			20	6020A
7440-70-2	Calcium	150000	480	96	mg/Kg			20	6020A
7440-47-3	Chromium	24	9.6	4.3	mg/Kg			20	6020A
7440-48-4	Cobalt	ND	1.9	0.72	mg/Kg			20	6020A
7440-50-8	Copper	6.1	9.6	3.8	mg/Kg	J		20	6020A
7439-89-6	Iron	190	48	19	mg/Kg			20	6020A
7439-92-1	Lead	ND	2.9	1.2	mg/Kg			20	6020A
7439-95-4	Magnesium	9600	480	240	mg/Kg			20	6020A
7439-96-5	Manganese	57000	48	19	mg/Kg			200	6020A
7440-02-0	Nickel	ND	4.8	1.9	mg/Kg			20	6020A
7440-03-1	Niobium	ND	24	12	mg/Kg			20	6020A
7440-09-7	Potassium	6700	96	38	mg/Kg			20	6020A
7782-49-2	Selenium	14	4.8	3.1	mg/Kg			20	6020A
7440-22-4	Silver	ND	1.9	0.72	mg/Kg			20	6020A
7440-23-5	Sodium	940	190	72	mg/Kg			20	6020A
7440-28-0	Thallium	ND	4.8	1.9	mg/Kg			20	6020A
7440-62-2	Vanadium	ND	9.6	3.8	mg/Kg			20	6020A
7440-66-6	Zinc	ND	48	19	mg/Kg			20	6020A

COVER PAGE
METALS

Lab Name: TestAmerica St. Louis Job Number: 160-23970-1

SDG No.:

Project: TAL Metals + Nb

Client Sample ID	Lab Sample ID
NFB-SLAG-01-170809	160-23970-1
NFB-SLAG-02-170809	160-23970-2
NFB-SLAG-03-170809	160-23970-3
NFB-SLAG-04-170809	160-23970-4
NFB-SLAG-05-170809	160-23970-5

Comments:

1A-IN
INORGANIC ANALYSIS DATA SHEET
METALS

Client Sample ID: NFB-SLAG-05-170809 Lab Sample ID: 160-23970-5

Lab Name: TestAmerica St. Louis Job No.: 160-23970-1

SDG ID.:

Matrix: Solid Date Sampled: 08/09/2017 12:00

Reporting Basis: DRY Date Received: 08/15/2017 08:30

% Solids: 99.9

CAS No.	Analyte	Result	RL	MDL	Units	C	Q	DIL	Method
7429-90-5	Aluminum	89000	46	18	mg/Kg			20	6020A
7440-36-0	Antimony	ND	4.6	1.8	mg/Kg			20	6020A
7440-38-2	Arsenic	ND	9.2	3.7	mg/Kg			20	6020A
7440-39-3	Barium	980	18	4.6	mg/Kg			20	6020A
7440-41-7	Beryllium	10	0.92	0.37	mg/Kg			20	6020A
7440-43-9	Cadmium	0.61	0.46	0.22	mg/Kg			20	6020A
7440-70-2	Calcium	140000	460	92	mg/Kg			20	6020A
7440-47-3	Chromium	160	9.2	4.1	mg/Kg			20	6020A
7440-48-4	Cobalt	0.80	1.8	0.69	mg/Kg	J		20	6020A
7440-50-8	Copper	93	9.2	3.7	mg/Kg			20	6020A
7439-89-6	Iron	640	46	18	mg/Kg			20	6020A
7439-92-1	Lead	9.3	2.8	1.1	mg/Kg			20	6020A
7439-95-4	Magnesium	20000	460	230	mg/Kg			20	6020A
7439-96-5	Manganese	560	4.6	1.8	mg/Kg			20	6020A
7440-02-0	Nickel	ND	4.6	1.8	mg/Kg			20	6020A
7440-03-1	Niobium	46	23	11	mg/Kg			20	6020A
7440-09-7	Potassium	ND	92	37	mg/Kg			20	6020A
7782-49-2	Selenium	14	4.6	2.9	mg/Kg			20	6020A
7440-22-4	Silver	1.7	1.8	0.69	mg/Kg	J		20	6020A
7440-23-5	Sodium	950	180	69	mg/Kg			20	6020A
7440-28-0	Thallium	ND	4.6	1.8	mg/Kg			20	6020A
7440-62-2	Vanadium	ND	9.2	3.7	mg/Kg			20	6020A
7440-66-6	Zinc	ND	46	18	mg/Kg			20	6020A

12-IN
PREPARATION LOG
METALS

Lab Name: TestAmerica St. Louis Job No.: 160-23970-1

SDG No.:

Prep Method: 3050B

Lab Sample ID	Preparation Date	Prep Batch	Initial Weight (g)	Initial Volume	Final Volume (mL)
MB 160-323386/1-A	08/21/2017 11:20	323386	0.5495		50
LCS 160-323386/2-A	08/21/2017 11:20	323386	0.5477		50
LCSSRM 160-323386/3-A	08/21/2017 11:20	323386	0.5372		50
160-23970-1	08/21/2017 11:20	323386	0.5389		50
160-23970-1 MS	08/21/2017 11:20	323386	0.5819		50
160-23970-1 MSD	08/21/2017 11:20	323386	0.5433		50
160-23970-2	08/21/2017 11:20	323386	0.5611		50
160-23970-3	08/21/2017 11:20	323386	0.5160		50
160-23970-4	08/21/2017 11:20	323386	0.5198		50
160-23970-5	08/21/2017 11:20	323386	0.5439		50

USEPA
DateShipped: 8/10/2017
CarrierName: FedEx
AirbillNo: 809412266698

CHAIN OF CUSTODY RECORD
Case #: 452
Contact Name: Smita Sumbaly
Contact Phone: 732-585-4410

17-08055
REC'D AUG 11 2017

No: 2-080917-182045-0012
Lab: Eberline Analytical
Lab Contact: Mike McDougal
Lab Phone: 865-481-0683

Lab #	Sample #	Sample Date	Sample Time	Analyses	Matrix	Lab QC	Numb Cont	Container
4	NFB-SLAG-01-170809	8/9/2017	12:00	Gamma Spec, Iso Th and U, Nb, TAL Metals	Soil/Rock/Slag	N	1	Ziploc Bag
5	NFB-SLAG-02-170809	8/9/2017	12:00	Gamma Spec, Iso Th and U, Nb, TAL Metals	Soil/Rock/Slag	N	1	Ziploc Bag
6	NFB-SLAG-03-170809	8/9/2017	12:00	Gamma Spec, Iso Th and U, Nb, TAL Metals	Soil/Rock/Slag	N	1	Ziploc Bag
7	NFB-SLAG-04-170809	8/9/2017	12:00	Gamma Spec, Iso Th and U, Nb, TAL Metals	Soil/Rock/Slag	N	1	Ziploc Bag
8	NFB-SLAG-05-170809	8/9/2017	12:00	Gamma Spec, Iso Th and U, Nb, TAL Metals	Soil/Rock/Slag	N	1	Ziploc Bag

Special Instructions: Gamma Spec(EPA 901.0). For Gamma Spec (homogenized) after 21 day ingrowth: Ra-226 using Bi-214 and/or Pb-214. From U decay chain: Th-234, Pa-234 or Pa-234m. From Th decay chain: Ra-228 and/or Ac-228, Ra-224, Pb-212, Bi-212, Tl-208. Other radionuclides: Cs-137, K-40. Alpha Spec: Iso U (EPA 908.0 Modified): U-233/234, U-235/236, U-238. Iso Th (EPA 907.0 Modified). Th-230, 232, 228. TAL metals plus Nb. All samples to be pulverized.

SAMPLES TRANSFERRED FROM

CHAIN OF CUSTODY #

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
5/Samples	WESTON	8/10/17 12:00		8-11-17 1100	Good
5/Samples	Bobby Bannister Eberline	8/14/17	TASR	8.15.17	

160-23970 Chain of Custody